

## UNITED STATES PATENT OFFICE

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## PROCESS OF LIBERATING PULP FROM RAW CELLULOSIC MATERIALS

No Drawing.

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This invention relates to the production of cellulose pulps from raw cellulosic materials such as wood, its object being to provide a two-step process of fiber liberation which may be advantageously applied in producing cellulose pulps of excellent physical and chemical characteristics. The product resulting from the process of the present invention is one contemplated for use both in the manufacture of high grade papers and in the preparation of cellulose derivatives, particularly if after liberation it is subjected to a bleaching and/or chemical refining process.

In producing pulps from wood by cooking in non-acid solutions of sodium sulphite, I have found that the resulting product is high in its pentosan content and low in tear resistance. Moreover, unless an alkali is present in the cooking liquor, it is necessary to go to much higher temperatures than those employed in usual chemical pulp-making process to effect fiber liberation. If the wood is precooked with water to hydrolyze and remove more especially the pentosans before the wood is cooked to complete fiber liberation in non-acid solutions of sodium sulphite, it is found that fiber liberation is completed with difficulty, especially if the precook in water is conducted at temperatures above 175° C., at which temperatures hydrolysis of pentosans is most effective, as disclosed in my co-pending application filed of even date herewith. So, too, the same difficulty is experienced when acid solutions are used for the precook short of fiber liberation,—for instance, acid sulphite solutions. Evidently the acid employed in the precooking liquor or organic acids generated in the water used in the precooking operation, while effective in causing a hydrolysis of the pentosans, at the same time causes a transformation of the ligneous content of the wood into reaction products difficult to dissolve with non-acid sulphite solutions, especially those containing no alkali or only a small amount of alkali.

I have discovered that a precooking of wood in dilute, non-acid sodium sulphite solutions is highly advantageous when fiber liberation is to be subsequently completed in non-acid sodium sulphite solutions of a strength requisite to the accomplishment of fiber liberation. Not only is such a precook effective in causing a removal of pentosans, but the quality of the resulting pulp, especially its tear resistance, is notably higher than when the precook is carried out in water or in acid solutions. An important operating advantage of the process of the present invention is that both the precooking and final cooking operations may be conducted in a single digester of the alkaline type, thus obviating the necessity of acid digesters and the transfer of the precooked material from one type of digester to another. Another advantage of my process is that, after the precook, sufficient sodium sulphite may be added to the hot, precooked mass of chips and liquor to complete fiber liberation, thereby conserving such residual or unspent sodium sulphite as is present in the liquor, as well as its heat content.

While alkalies such as caustic soda may be added to the precooking solution of sodium sulphite, it is preferable to operate with a straight monosulphite solution, as such a solution is most effective in removing pentosans from the wood. The second cooking operation to complete fiber liberation may, however, be advantageously performed in a solution containing an alkali such as caustic soda, as the function of the second cooking operation is more especially to remove ligneous matter from the wood, for which purpose alkali is highly effective. In other words, the precooking operation having accomplished the desired removal of pentosans from the wood, the purpose of the second cooking operation should be, as it is in accordance with the present invention, the removal of sufficient ligneous matter from the wood to cause fiber liberation into a pulp of high alpha cellulose content.

I shall now give specific examples of procedure falling within the purview of the present invention. Spruce chips of a size such as used in customary chemical pulp-making processes were placed in a digester with a 1% solution of sodium sulphite. The digester was closed and its contents were heated to 350° F., which temperature was

maintained for three hours. Upon examination of the chips after this initial cook, it was found that they were still hard and showed no sign of pulping. The chips were then digested for five hours at 370° F. in another liquor containing 10% sodium sulphite and 1% caustic soda to produce pulp. The resulting pulp, when washed free of cooking liquor and tested, was found to have an alpha cellulose content of 95.6%, a pentosan content of 1.9%, a tear resistance of 270, and a strength of 147. Another procedure was carried out under conditions similar to those given in the previous example, except that a straight 10% sodium sulphite solution was employed as the second cooking liquor. The resulting product was practically similar to that of the previous example, except that it had an alpha cellulose content of 93 and a strength of 175. This indicates that the presence of a small amount of caustic soda in the second cooking liquor favors the production of a product of high alpha cellulose content but of somewhat lower strength.

If only the second cooking operation is performed on the raw chips, it is found that the resulting pulp is much poorer in its all-round characteristics. Thus, the product from such a single cooking operation has an alpha cellulose content of 87%, a pentosan content of 9%, a tear resistance of 160, and a strength of 180. This clearly demonstrates the value of a precook in accordance with the present invention.

While the foregoing specific examples of procedure have to do with the use of spruce wood as the raw cellulosic material, the present invention may be applied to advantage in the case of other raw cellulosic material, for instance hardwoods, which have a pentosan content higher than spruce wood. In any event, however, the two-step cooking procedure of the present invention performs the same general functions, although the characteristics of the pulps resulting under a given set of cooking conditions may vary more or less, depending upon the kind of raw cellulosic material used. When the cooking liquor is allowed to remain along with the chips in a hot condition in the digester after the initial cooking operation, the sodium sulphite necessary to strengthen the liquor to the desired concentration for the second cooking operation may be injected into the digester as a concentrated solution of sodium sulphite, containing, if desired, some alkali. The temperature of the digester contents may then be raised, if the second cooking operation is to be performed at higher temperatures than the initial cooking operation.

What I claim is:

1. A process which comprises cooking raw cellulosic material in a dilute non-acid sulphite solution, and then cooking to complete

fiber liberation in a more concentrated non-acid sulphite solution.

2. A process which comprises cooking raw cellulosic material in a dilute non-acid sulphite solution, adding sulphite to the hot mass to increase concentration of the solution, and cooking to complete fiber liberation in the more concentrated solution.

3. A process which comprises cooking raw cellulosic material in a dilute, substantially neutral sulphite solution, and then cooking to complete fiber liberation in a more concentrated sulphite solution containing alkali.

4. A process which comprises cooking raw cellulosic material in a dilute, substantially neutral sulphite solution, and then cooking to complete fiber liberation in a more concentrated sulphite solution containing a relatively small proportion of alkali.

5. A process which comprises cooking raw cellulosic material in a substantially neutral 1% solution of sodium sulphite, and then cooking to complete fiber liberation in a more concentrated, non-acid, sodium sulphite solution.

6. A process which comprises cooking raw cellulosic material in a substantially neutral 1% solution of sodium sulphite, and then cooking to complete fiber liberation in a non-acid sulphite solution of at least about 10% strength.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.